CHEMICAL COMPOSITION OF MICROPLASTICS FLOATING ON THE MEDITERRANEAN SEA SURFACE

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Abstract – The Mediterranean Sea is one of the most studied regions in the world in terms of microplastic (MP) pollution. With an estimated input of plastic of approximately 100 kt per year, it is considered as one of the largest hotspots of plastic litter accumulation in the world [1]. However, the observation of a consistent distribution and concentration pattern is difficult. Indeed, microplastic investigations in the Mediterranean Sea surface often concern localized areas. Moreover, the different studies are uneasy to compare due to the heterogeneity of the Mediterranean Sea environment and the variety of methodologies used. In addition, only a few studies have analysed the chemical composition of microplastics at the Mediterranean Sea surface. In this context, the main objective of the present study is to describe the chemical composition of MP collected in the surface waters of the Mediterranean Sea, as well as the size of the particles [2].
Our results pointed to a certain homogeneity at the Mediterranean Sea scale. The main polymers collected were polyethylene (PE) (67.3±2.4 %), polypropylene (20.8±2.1 %) and polystyrene (3.0±0.9 %). Nevertheless, discrepancies, confirmed by the literature, were observed at a mesoscale level. Thus, in the North Tyrrhenian Sea, the proportion of PE was significantly lower than the average value of the Mediterranean Sea (57.9±10.5 %). In congruence with the current state of knowledge, different hypotheses are proposed to explain these discrepancies: anthropic sources, rivers, horizontal and vertical distribution phenomena.

Introduction

The Mediterranean Sea is a semi-enclosed sea with densely populated coasts that concentrates miscellaneous intensive marine and terrestrial activities and receives water from important river catchments (e.g. Nile, Ebro, Rhône and Po). It is therefore no coincidence that it has been reported to be one of the largest hotspots of plastic litter accumulation in the world with an estimated input of plastic of approximately 100 kt per year [1], [3].

These plastic particles are composed of large proportions of microplastics (MP) ranging from millimetre-sized to micrometre-sized particles [4]. MP are found to be present

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in every studied Mediterranean shoreline and island from 18 coastal countries [5]. Despite an important spatial and temporal variability, highest microplastic concentrations are found to be near the more densely populated coastlines [6]. Statistical calculations have estimated that more than 75% of floating plastics reside in the 50 km near-shore waters [7]. Moreover, a segregation of plastic types with increasing distance to shore has been observed [6]. The detected plastic types are diverse but some are predominant on the sea surface because of their widespread use and their buoyancy: polyethylene (PE), frequent in food packaging (e.g. in films and bottle caps); polypropylene (PP), used as packaging material and plastic parts in various industries; polyamides (PA) and polystyrene (PS) [8].

Despite a growing number of papers, knowledge is still lacking to fully understand the distribution and concentration and chemical composition of MPs on the surface of the Mediterranean Sea [9]–[14]. Furthermore, the different studies are uneasy to compare because of the heterogeneity of the Mediterranean Sea environment (hydrodynamic features, seasonality) and the variety of methodologies used (e.g. sampling, microplastic extraction, analysis, sizes considered, concentration units) [10].

In this context, where a global vision at the scale of the Mediterranean Sea was missing, the Tara Ocean Foundation, a French non-profit organisation dedicated to the study of the world's oceans, carried out microplastic samplings for 5 months in 2014 across the entire Mediterranean Sea. This expedition thus made it possible to map various areas using the same methodologies.

This poster and proceeding are an excerpt from our work recently published in the Marine Pollution Bulletin journal [2]. This publication aimed to quantify (concentrations in mass and in number) and qualify (size and chemical nature) microplastic pollution at the surface of the Mediterranean Sea. Here, only the results concerning the chemical composition and sizes of the collected microplastics are presented.

Materials and Methods

Microplastic samples were collected in Mediterranean Sea waters during the Tara Mediterranean Expedition between May and November 2014 (Fig. 1). Using a 333µm-mesh size manta net (net opening: 16x60 cm), samplings were carried out in surface waters in 124 sites across the Mediterranean Sea.

In the laboratory, plastic debris were carefully separated from the other components (plankton, wood, etc.) under a dissecting microscope. Plastic particles were counted and measured using a ZooScan digital scanner at the Laboratoire d'Océanographie de Villefranche-sur-Mer (LOV, Villefranche-sur-Mer, France) [6]. Plastic samples were then transferred to the Ifremer LERPAC laboratory (France) to be weighed before chemical analysis. A total of 15,654 particles from 54 selected sites were wet sieved by size class (]5-2 mm],]2-1 mm],]1-0.5 mm],]0.5-0.315 mm]), sorted and transferred to 96-well microplates and named with a unique identifier at the Institut de Recherche Dupuy de Lôme (IRDL, Lorient, France). A statistical approach based on a random draw of particles was used to limit the amount of work required to analyse the MP by infrared spectroscopy (ATR-FTIR). The aim of this method was to analyse only a statistically representative proportion of the total population of microplastics collected [15]. MP spectra were acquired using an Attenuated Total Reflection Fourier Transform Infrared spectrometer (ATR-FTIR Vertex70v,

Bruker). All spectra were recorded in absorbance mode in the 4000–600 cm^{-1} region with a resolution of 4 cm^{-1} and 16 scans. All the spectra were then identified using the POSEIDON (Plastic pOllutionS ExtractIon, DetectiOn and aNalysis) software [16] which was developed with R i386 3.1.2.

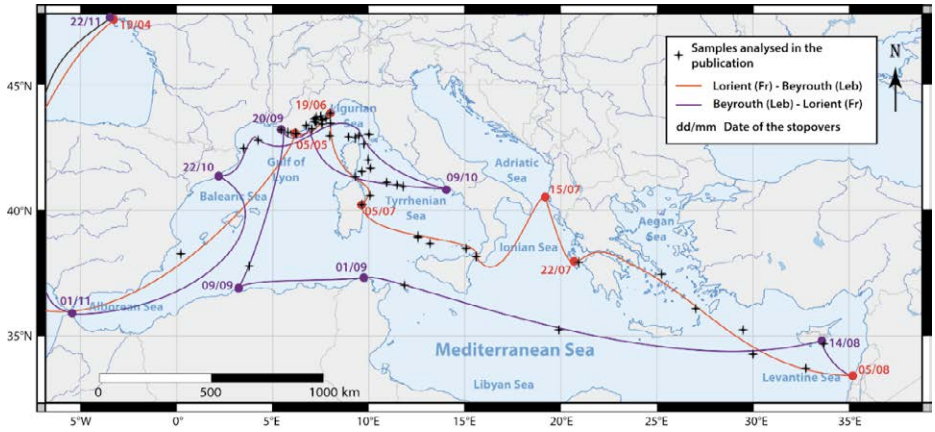

Figure 1 – Routes of the Tara schooner during the TARA Mediterranean Expedition in 2014.

Results

Overview of the microplastic pollution in the Mediterranean Sea and its sub-basins

At the Mediterranean scale, the collected particles were mainly PE (67.3 ± 2.4 %), PP (20.8 ± 2.1 %), and PS (3.0 ± 0.9 %) (Fig. 2.a.). The other identified polymers (i.e. polyethylene vinyl acetate, ethylene propylene rubber, poly(methyl methacrylate), polyamide, polystyrene, polyurethane, polyvinyl chloride, and polyethylene and polypropylene like) represented 6.0 ± 1.2 % of the total sampled particles. For the three sub-basins studied (Gulf of Lyon, Tyrrhenian Sea and Eastern Mediterranean basin) the distribution of polymer types was similar to the one observed at the Mediterranean Sea scale. Nevertheless, in the Tyrrhenian Sea, the PP rate was significantly higher than the average value of the Mediterranean Sea. In the Eastern Mediterranean basin, the data showed a significantly lower rate of the category “other polymers”.

The proportions of the different chemical natures highlighted in this study varied significantly according to the particle size range (Fig. 2.b). Thus, the PE content of the samples was 76.1 ± 7.0 % for particles between 2 and 5 mm, and fell to only 38.3 ± 6.4 % in the 315 to 500 µm particle size range. PS showed a similar tendency, with proportions of 7.7 ± 4.4 % to 0.5 ± 0.9 % respectively. Conversely, the PP content of the samples increased as the particle size range decreased. Indeed, PP represented 10.6 % of the particles for particles between 2 and 5 mm, and increased up to 49.1 ± 6.6 % in the particle size range from 315 to 500 µm.

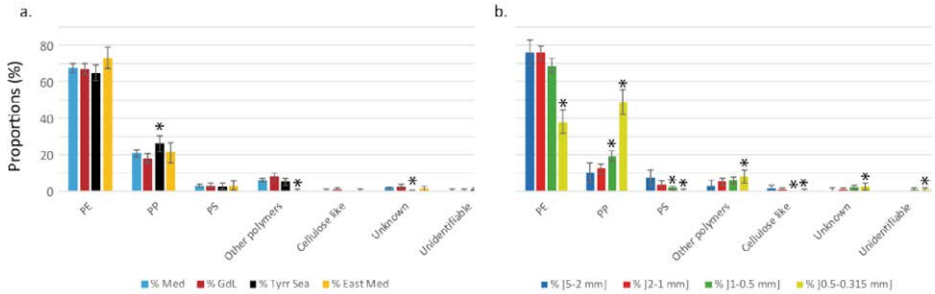

Figure 2 – Proportion of the chemical natures of the collected MP (±Interval of confidence). a. Proportions of the different chemical natures at the Mediterranean scale (Med) as well as for the three sub-basins studied: The Gulf of Lyon (GdL), the Tyrrhenian Sea (Tyrr Sea) and the eastern Mediterranean basin (East Med) (±Interval of confidence). The chemical composition varied little from one basin to another. Only the Tyrrhenian Sea had a significantly (*) higher PP content than the Mediterranean Sea. b. Proportions of the different chemical natures according to the size range of the collected particles (±Interval of confidence). Statistically significant variations (*) of the chemical nature were evidenced depending on the size range studied.

Discussion

Chemical composition of MP on the surface of the Mediterranean Sea Comparison with previous work and limitations

Few studies have analysed more than a few hundred MP in the Mediterranean. Among them, Baini *et al.* (Tuscany, Italy) and Zeri *et al.* (Adriatic Sea) have shown chemical composition results very similar to those of our study [9], [17]: PE rates of about 66.0 ± 5.2% and 66.5 ± 2.6% respectively. On the contrary, other studies have shown different results, with notably lower PE rates: 41.2 ± 3.3 % in the south of the Adriatic Sea [18], 60.0 ± 1.9 % in the north of this sub-basin [12], 55.0 ± 1.7 % and 43.0 ± 4.6 % in the western basin of the Mediterranean Sea [11], [18]. The results of these two publications differ significantly from those of our publication. In contrast, two other studies have evidenced high percentages of PE in the range of 76 ± 3.9 to 79 ± 5.4 % on the southern coasts of the Mediterranean Sea [13], [14]. Nevertheless, these values are consistent with those found by the present study (73.1 ± 5.8 %) in the eastern Mediterranean Sea basin. Finally, it is interesting to note that the PE rate in the Mediterranean Sea is on average higher than in the Atlantic ocean (<60 %) [19].

However, the comparison of the data obtained between our study and these publications presents various limitations [2], [9], [11], [12], [17], [18]. The first is undoubtedly linked to a scale effect. Indeed, the smaller the sampling in terms of number of sampling points, the more sensitive it will be to local variations. Thus, it may be more prone to deviate from the average value for the Mediterranean Sea. However, the lack of data leads to the comparison of studies carried out at different spatial scales. The use of statistical calculations considering the number of particles analysed (i.e. calculation of the interval of confidence, proportion test) allows to partially compensate this bias of geographical scale.

Furthermore, the lack of standardized sampling methods is a hindrance when it comes to comparing MP studies with each other [10]. The influence of the net mesh size is assumed to be crucial in MP size measurements. For example, sampling in the Seine River with an 80 µm net yields on average 30 times more MP (in numbers) than sampling with a 333 µm net [20]. As it can differ between publications, discrepancies in MP concentrations, but also in PE proportions, could be induced by this parameter.

Chemical composition and size range

Our results evidenced an increase in the PP rate with decreasing particle size. A similar trend has been demonstrated in the Mediterranean Sea by Baini *et al.* [9], but also in plastic samples from different oceans [21]. In fact, this trend could be explained by a higher ageing sensitivity of PP than PE, enabling PP to fragment into small dimensions faster [22]. The PE/PP ratio therefore seems to be an interesting data to characterise plastic pollution and may be an indicator of a segregation phenomenon undergone by plastic pollution on the ocean surface.

Variations observed at smaller geographical scale: the case of the North Tyrrhenian Sea

If our study tends to highlight differences in polyethylene levels, the analysis of previous scientific publications also seems to show some similar trends.

In the North Tyrrhenian Sea, the PE and PP proportions were respectively abnormally low and high (Fig. 3) compared to other sampled areas in the North Mediterranean Sea (Fig. 3). This lower proportions of PE in the North Tyrrhenian Sea could imply the existence of local sources introducing MP pollution with low levels of PE (i.e. <65 % of PE). These MP can come from four main sources: rivers, cities, maritime traffic and sea currents [7], [23] (Fig. 3). There are currently no data for the Italian rivers flowing into the Tyrrhenian Sea. Based on data further away from this geographical framework, it appears that the composition of MP pollution in European rivers is generally poor in PE and rich in PP and PS. Indeed, PE proportions have been reported to be quite low in samples collected on the surface of lakes tributary of the Po and in the delta of the Rhône river (RR; Fig. 3) but also in north European rivers (without connection to the Mediterranean Sea) (<50 %) [1], [24], [25].

Furthermore, the relatively high average size of the MPs collected in the north of the Tyrrhenian Sea can possibly be interpreted as an indication of a source of microplastic relatively close to the sampling areas. Cities such as Rome, Naples or Livorno are other potential important sources of MP [7], [23]. However, data are very scarce in the case of Mediterranean cities and, more generally, in the case of this kind of proximal sources that therefore need to be better studied [11]. In other locations around the world, MP observed in cities (i.e. water and soils) are heterogeneous and have rather low PE contents [26]–[28].

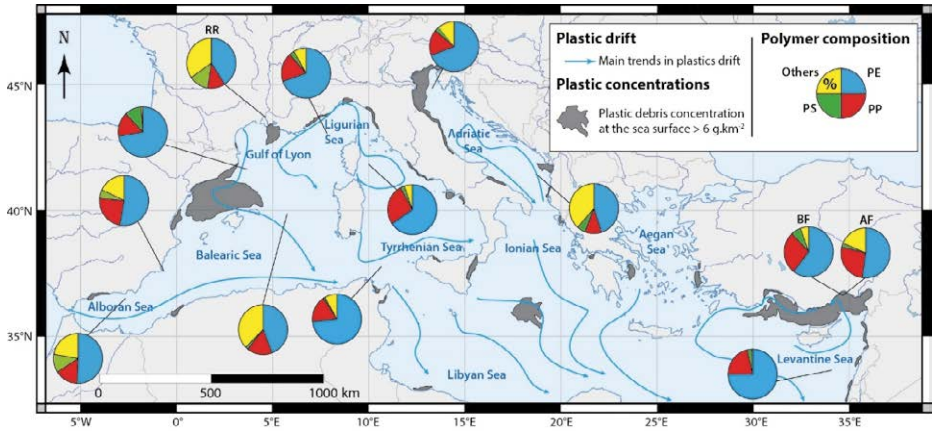

Figure 3 – Synthesis map of the sources, drift, concentration and composition of MP in the Mediterranean Sea. Data on concentrations and drifts of MP are based on numerical modelling [7]. The data on chemical types are taken from the current study and various other publications ([9], [11]–[14], [18], [29]). RR: Rhône river; BF: Before flooding; AF: After flooding.

Spectral variability of microplastics collected in the Mediterranean Sea: example of PE spectra

Many FTIR spectra of Tara samples presented additional bands, in particular between 1200 and 800 cm^{-1} (attributed to biofouling), and between 1800 and 1500 cm^{-1} (attributed to polymer weathering by oxidation). These bands are assumed to be induced by marine environment weathering [30]–[32]. These new bands can lead to errors when spectra of weathered polymers are compared in an automated way with reference databases of virgin polymers [32].

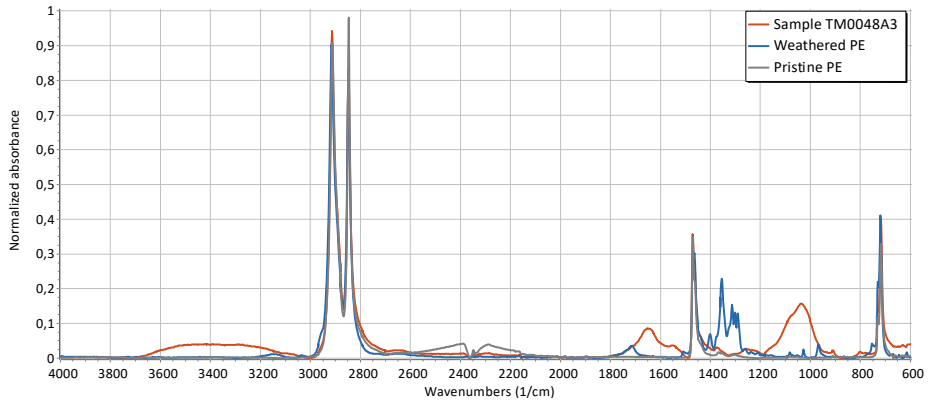

Figure 4 – Infrared spectra of pristine PE (gray), artificially UV-weathered PE (blue) and a PE microplastic from the Tara mission (TM0048A3) (orange). The band between 1100 cm^{-1} and 1045 cm^{-1} could reflect the presence of diatoms on the surface of TM0048A3.

For example, in the case of the TM0048A3 sample, a PE pellet, it was possible to observe on its surface areas of green colour (Fig. 5). SEM observation revealed the presence of diatoms (*Bacillariophyta*) that had largely colonised the anfractuosities (Fig. 5). Analysis of the FTIR spectrum showed that in addition to the band at 1045 cm^{-1}, a characteristic shoulder is visible at 1100-1060 cm^{-1}, reflecting the presence of diatom frustule silica (Fig. 5) [33]. This diatom fingerprint was visible on a very large number of PE spectra. Thus, from the point of view of FTIR spectra, diatoms could be a good marker of the plastisphere since the study of microalgae by FTIR is well established in the literature [33].

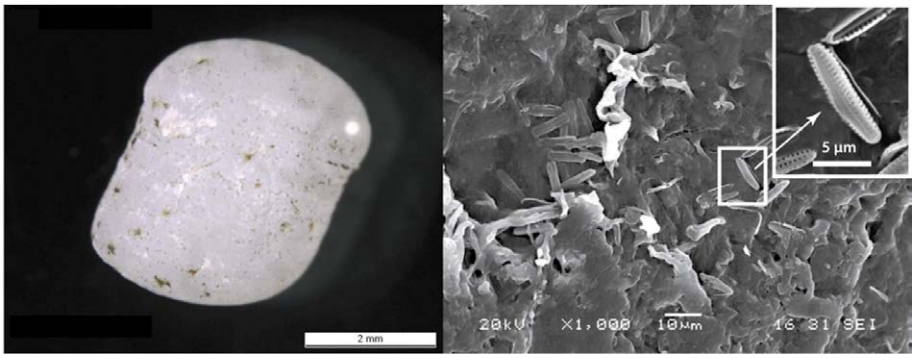

Figure 5 – SEM photograph of the surface of the sample TM0048A3. Diatoms are clearly visible.

Conclusion

The objective of this work was to evaluate the chemical nature of the microplastic pollution at the surface of the Mediterranean Sea. The samples were collected by manta net during the Tara Mediterranean expedition, carried out between June and November 2014. Microplastics from 54 sites were analysed by FTIR spectroscopy, and size, concentrations in mass and in number were measured. At the Mediterranean scale, the collected particles were mainly PE (67.3 ± 2.4 %), PP (20.8 ± 2.1 %), and PS (3.0 ± 0.9 %). The results point towards a certain homogeneity of the chemical nature of microplastics in the Mediterranean during the sampling period. However, differences, confirmed by the literature, were observed at a mesoscale level. New studies involving more geographically targeted samplings, temporal monitoring or numerical modelling are needed to confirm, invalidate or refine some of the hypotheses made here. Finally, further investigations are needed to explore the potential of infrared spectroscopy to study the fouling of microplastics at sea.

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